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Dentistry — Test methods for tensile bond strength to tooth structure

Médecine bucco-dentaire — Méthodes d'essai pour l'adhérence par traction à la structure dentaire

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Foreword

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This document was prepared by Technical Committee ISO/TC 106, *Dentistry*, Subcommittee SC 1, *Filling and restorative materials*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Adhesion in restorative dentistry is an important topic. It is the intention of this document to describe various laboratory procedures of tensile bond strength tests whereby the effect or quality of a bond between a dental material and tooth structure can be substantiated.

Adhesive materials are used in many types of restorative and preventive treatments. Even if the stress on the bond in most circumstances can be defined as either tensile, shear or a combination of these, there are no specific laboratory tests which can be valid for all the various clinical applications of adhesive materials.

The relative performance of materials that are claimed to bond to tooth structure is usually evaluated by laboratory assessment of bond strengths. While bond strengths are unable to predict clinical behaviour or performance, they are useful for comparing adhesive materials.

ISO 29022[1] prescribes the notched-edge shear bond strength test.

Annex A lists several published laboratory methods for tensile bond strength measurement.

Tensile bond strength testing is also common in general materials science, and a publication listing many test methods is provided for information in Reference [2].

Dentistry — Test methods for tensile bond strength to tooth structure

1 Scope

This document gives guidance on substrate selection, storage and handling as well as essential characteristics of tensile bond strength test methods for quality testing of the adhesive bond between restorative dental materials and tooth structure, i.e. enamel and dentine. Some specific test methods for tensile bond strength measurements are given in Annex A.

This document does not include requirements for components of adhesive materials and their performance.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 1942, *Dentistry — Vocabulary*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 6344-3, *Coated abrasives — Determination and designation of grain size distribution — Part 3: Microgrit sizes P240 to P5000*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 1942 and the following apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
adhere
to be in a state of *adhesion* (3.3)

3.2
adherend
body that is held or intended to be held to another body by an *adhesive* (3.4)

3.3
adhesion
state in which two surfaces are held together by chemical and/or physical forces with the aid of an *adhesive* (3.4)

3.4
adhesive
substance capable of holding materials together

3.5
bond strength
force per unit area required to break a bonded assembly with failure occurring in or near the *adhesive* (3.4)/*adherend* (3.2) interface

3.6
substrate
material upon the surface of which an *adhesive* (3.4) is spread for any purpose, such as bonding or coating

4 Sampling

The amount of test material shall be sufficient for all planned tests and be from the same batch.

5 Test methods

5.1 General

Adhesive materials are used for many different purposes in the oral cavity. This document describes tensile bond strength tests. When bond strength is to be measured, the raw data are in unit of force (N). It is necessary to convert this into stress unit, i.e. force per unit area (MPa). Hence, control of the area and roughness of the surface for application of the adhesive material are important.

Two critical variables or factors shown below shall be considered when designing test equipment and preparing specimens for tensile testing of bond strength:

a) alignment of the tensile forces acting on the specimen;

b) limitation of the bonding area.

5.2 Tooth substrate and storage

5.2.1 Substrate

5.2.1.1 Types of the substrate

Use either human permanent premolars/molars or bovine mandibular incisors for the measurement of bond strength. The donor bovine shall not be more than five years old.

5.2.1.2 Characteristics of the substrate

When measuring bond strength to human dentine, this document recommends to use the buccal superficial dentine that is as close to the enamel as possible in order to reduce variations. It is preferable to use third permanent molars from 16-year-old to 40-year-old individuals if possible.

5.2.2 Time after extraction

There is increasing evidence that changes in dentine occur after extraction that can influence bond strength measurements. The effect can vary with different types of bonding materials. Ideally, bond strengths should be measured immediately post-extraction.

NOTE Teeth within six months after extraction can be used. Teeth extracted more than six months prior to use undergo degenerative changes in dentinal proteins.

5.2.3 Condition of teeth

5.2.3.1 History on clinical treatment (of human teeth)

Human teeth used for bond strength measurement shall be caries-free and preferably unrestored. However, small and superficial restorations not in the adhesion test area are acceptable. Root-filled teeth shall not be used.

5.2.3.2 Site of the dentition and life-history of the donating patient (of human teeth)

There is some evidence to suggest that different teeth in the dentition can give different results with bonding to dentine and enamel. It is not possible to have complete control of variables such as age of the donating patient, ethnicity and dietary history, state of health, or to standardize the composition and structure of the teeth.

5.2.4 Preparation and storage of teeth

5.2.4.1 Preparation of extracted teeth

Immediately after extraction, human teeth shall be thoroughly washed in running water and all blood and adherent tissue removed, using sharp hand instruments. Bovine teeth shall be cleaned as soon as possible after extraction and the soft tissue in the pulp chamber shall be removed in a similar fashion.

5.2.4.2 Storage of prepared teeth

Prepared teeth shall be placed in distilled water of grade 3 in accordance with ISO 3696 or in a 1,0 % chloramine-T trihydrate (CAS Registry Number®[1] 7080-50-4) bacteriostatic/bacteriocidal solution for a maximum of one week, and thereafter stored in distilled water grade 3 in a refrigerator, i.e. nominal 4 °C. To minimize deterioration, the storage medium shall be replaced at least once every two months.

It is essential that no other chemical agents be used, as they can be absorbed by tooth substance and alter its behaviour.

NOTE Chloramine-T is a strong oxidant and can oxidise polymerisation initiators, particularly reducing agents in chemical polymerisation initiator systems. The oxidation reaction affects the chemical polymerisation by redox reaction between the oxidising agent and reducing agent and can reduce bond strength.[13]

5.3 Tooth surface preparation

5.3.1 Requirement for the surface of tooth to be adhered

A reproducible flat surface is required. Tooth surfaces shall be kept wet at all times during preparation.

NOTE Exposure of the surface of prepared tooth to the air for several minutes can cause irreversible changes in bonding character. Dentine is especially sensitive to dehydration.

1) Chemical Abstracts Service (CAS) Registry Number® is a trademark of the American Chemical Society (ACS). This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

5.3.2 Requirement for the procedure of preparation of tooth surface

5.3.2.1 Blocking of pulp chamber (for bovine teeth)

The pulp chamber of bovine teeth shall be blocked, for example by wax, to prevent penetration of resin into the dentine. Alternatively, use a high viscosity potting medium that does not penetrate the pulp chamber.

NOTE This can be verified by preparing a set of potted teeth and examining the pulp chambers for the presence of polymerised resin.

5.3.2.2 Retention form in test tooth

Ensure that the tooth has form (undercuts, holes or retentive pins) that will secure retention in the mounting medium.

5.3.2.3 Mounting the prepared tooth in a holder

To control the planing and the angle of the surface during preparation, the test tooth shall be mounted and immobilised in a rigid holder by means of dental die stone or low exothermal-curing viscous resin. The temperature of the polymerising resin shall not exceed 42 °C.

NOTE The absorption of resin can adversely affect the tooth.

5.3.2.4 Storage of the mounted tooth

Place the mounted tooth in water at (23 ± 2) °C as soon as possible.

5.3.3 Surface preparation of tooth plane for adhesion

5.3.3.1 Procedure of planing

a) A standard surface shall be prepared by planing the exposed surface of the tooth against silicon carbide abrasive paper finally with a grit size of P600 as defined in ISO 6344-3 fixed on a hard and flat plate under running water.

   NOTE The "P" prefix by the grit number means this coated abrasive conforms to the Federation of European Producers of Abrasives (FEPA)[2)] grading system.

b) Grind until the surface is even and smooth.

5.3.3.2 Inspection and treatment

a) When inspected visually, the surface shall be even and smooth.

b) Discard teeth that have perforations into the pulp chamber.

c) Discard teeth that have craze lines and cracks. Inspection can be carried out under light microscopy and external fibre optic lighting.

d) Ensure that the surface is confined to enamel or dentine.

2) https://fepa-abrasives.org/

5.4 Application of adhesive

5.4.1 General

5.4.1.1 Ambient conditions

All of the procedures shall be performed at (23 ± 2) °C and (50 ± 10) % RH.

5.4.1.2 Preconditioning of the prepared surface of the tooth

The tooth surface prepared for application of adhesive material shall be preconditioned according to the manufacturer's instructions. If no instructions are given, rinse with running water for 10 s and remove visible water on the surface with a filter paper or by a light/brief stream of oil-free compressed air immediately before application of the adhesive material.

5.4.1.3 Application of the adhesive material

Mix if necessary, and apply the adhesive material according to the instructions given by the manufacturer.

5.4.2 Adhesive and/or adherend material in bulk

5.4.2.1 Importance of limitation of the bonding area

A limitation of the bonding area is an important consideration.[3]

5.4.2.2 Methods to limit the bonding area

5.4.2.2.1 General

The typical examples of the methods to limit the bonding area are to use a material holder or a double sided-adhesive tape.

5.4.2.2.2 Material holder to limit the bonding area

For example, limiting the bonding area can be achieved by using a material holder with a sharp edge contacting the tooth surface. This holder is also able to stabilise the material(s) on the tooth surface for curing.

This holder also allows restriction of the substrate treatment area and demarcation of the extent of the adhesive and permits accurate limiting of the bonded surface.

5.4.2.2.3 Material holder for light-curing adhesive or adherend materials

a) The transparency of the material holder shall be such that, for light-curing adhesives or adherend materials, the material holder shall give sufficient access to the curing light to allow a sufficient amount of the light (e.g. by being made partly or totally of a transparent material).

b) The amount of light energy applied to the material shall be in accordance with the manufacturer's instructions.

5.4.2.2.4 Procedure to use the material holder

a) When using the same material holder several times, coat the inner part of the material holder with a mould-releasing agent. Avoid coating the edge of the holder.

NOTE If the edge of the material holder is coated with the mould-releasing agent, the agent can contaminate the surfaces to be adhered and therefore will affect the adhesion.

b) Apply a thin layer of the adhesive material onto the holder surface.

c) Fill the material holder to slight excess with the adhesive or the adherend material and place it firmly in the correct position on the tooth.

d) Ensure that the material holder maintains contact with the tooth surface in the correct alignment during fixation.

e) If the manufacturer recommends a particular restorative material for use with the adhesive under investigation, then the same restorative material shall be used for all tests of that adhesive.

f) The fixation of the material holder shall be finished within the manufacturer's stated working time of the adhesive material.

5.4.3 Adhesive material as thin film and adherend material as preformed rod

a) If it is decided to restrict the bonding area and use an adherend rod, fix a thin tape of material that is non-reactive with the adhesive with a hole of the same dimensions as the contact area of the rod, to the planed tooth surface.

b) Apply a thin layer of the adhesive material on the tooth surface inside the hole in the tape and lower the adherend rod to contact the adhesive material inside the hole.

c) Fix the rod in exact position and alignment and place a constant load on top for constant time.

The rod should have flat top- and bottom-surfaces, and they should be parallel to each other. To facilitate the application of the load evenly, the rod should be placed in a position normal to the tooth surface where the adhesive has been applied.

d) The total procedure from application of the material to the fixation of the upper rod shall be performed within the manufacturer's stated working time.

e) Remove the tape after curing without applying any adverse force on the bonded specimen.

5.5 Storage of test specimens

5.5.1 General

5.5.1.1 Temperature

Test specimens shall be prepared at (23 ± 2) °C and stored in water at (37 ± 2) °C prior to testing.

NOTE Storage in water for 24 h is normally sufficient to discriminate between materials that can withstand wet environment and those that do not.

5.5.1.2 Thermocycling

Thermocycling between 5 °C and 55 °C may be used as an accelerated ageing test.

5.5.1.3 Simple storage in water

Longer periods of water storage are supposed to be necessary to assess durability of the bond.

NOTE Simple water storage has been found to mimic clinically observed degradation of restorations.[12],[14]

5.5.2 Recommended procedures and conditions of durability test

5.5.2.1 Treatment of adhesion test specimen in water

a) Test type 1: short-term test for 24 h in water at 37 °C.

b) Test type 2: thermocycling test comprising 500 cycles in water between 5 °C and 55 °C, starting after (20 to 24) h storage in water at 37 °C.

   The exposure to each bath should be at least 20 s, and the transfer time between baths should be (5 to 10) s.

c) Test type 3: long-term test after six months storage in water at 37 °C (medium changed every 7 d to avoid contamination).

5.5.2.2 Timing of the tensile test after the treatment in water

The specimens shall be tested for bond strength immediately after removal from water.

5.6 Apparatus

5.6.1 General

Several types of apparatus are available for measuring the tensile bond strength of an adhesive system. The critical requirements for selection of a suitable instrument for the small and sometimes fragile specimens are:

a) the ability to mount the tooth/material specimen in the apparatus and the universal testing machine without application of load (tensile, bending, shear or torsion) on the specimen;

b) a sufficiently rigid construction, in order to avoid elastic deformation (or displacement) of the apparatus and the connection to the testing machine;

c) the ability to apply a slowly increasing and unidirectional tensile load, and the ability to align the specimen to avoid an uneven stress distribution during loading.

5.6.2 Alignment

The test apparatus shall secure alignment between substrate and adhesive material, i.e. the tensile force shall be applied at a 90° angle to the planed substrate surface.

5.6.3 Connecting device

The connection between the apparatus and the crosshead of the universal testing machine shall be by a universal joint, chain or wire.

5.7 Tensile loading

a) Perform the test at (23 ± 2) °C and (50 ± 10) % RH.

b) Mount the tensile test specimen in the testing apparatus. Do not apply any bending or rotational forces to the adhesive material during mounting.

c) Apply the tensile load as described in 5.8.

5.8 Test

The standard crosshead speed for testing a bonded specimen is recommended to be (0,75 ± 0,30) mm/min.

5.9 Recording, treatment and analysis of results

5.9.1 Recording and treatment of results

5.9.1.1 Debonding before tensile test

Unless clearly due to specimen mis-handling, it shall be ascribed a bond strength value of zero (0,0) MPa.

5.9.1.2 Treatment of results

Record the maximum force (N) prior to failure of the bond. Calculate the bond strength (stress) using Formula (1):

$$\sigma = \frac{F}{A_b} \tag{1}$$

where

σ is stress, expressed in MPa;

F is force, expressed in N;

A_b is bonding area, expressed in mm^2.

5.9.2 Analysis of results

The bond strength values obtained by tensile testing generally show large coefficients of variation, i.e. (20 to 50) %, and shall be analysed statistically by an appropriate method.[4]

If the coefficient of variation is above 50 %, a thorough inspection of the overall procedure is recommended.

5.10 Report

Report the following:

a) the substrate used (human or bovine teeth, enamel or dentine);

b) the tooth preservative (type of medium and the temperature applied) and disinfection conditions (if any);

c) the storage temperature of teeth;

d) the storage period of the teeth;

e) the adhesive used, lot number and expiry date;

f) the material in bulk or preformed rod used as adherend, lot number and, if applicable, expiry date;

g) the curing light used and curing mode, if applicable (e.g. lamp type, pulse, intensity);

h) the individual bond strength values (MPa), sample size (i.e. number of specimens tested), mean and standard deviation;

i) the identity of the test lab;

j) the date of the tensile test;

k) the date of the report prepared.

Annex A
(informative)

Test methods for measurement of tensile bond strength

A.1 General

Annex A lists several examples of published test methods for measurement of tensile bond strength to tooth structure with a short description of principles and the reference(s) to publications for a more complete description, respectively.

A.2 Test methods

A.2.1 Kemper and Killian's test method[5],[6]

A.2.1.1 Jigs and alignment

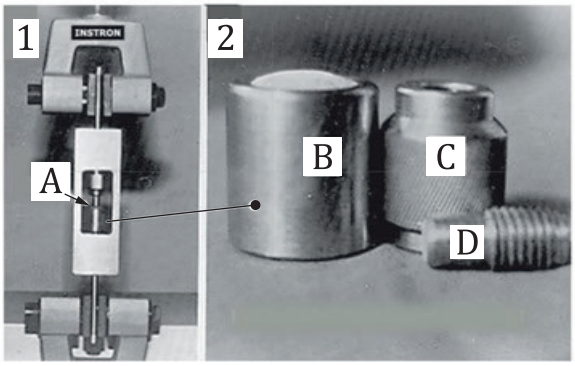
Jigs for this test method consists of a multi-part test apparatus to ensure alignment during specimen preparation and testing as below (see Figures A.1 and A.2):

a) holders for material and the tooth, respectively (tooth cup, material cup);

b) a bonding alignment block;

c) a set of rods for the connection to the universal testing machine;

d) translucent holders especially for light-curing materials.



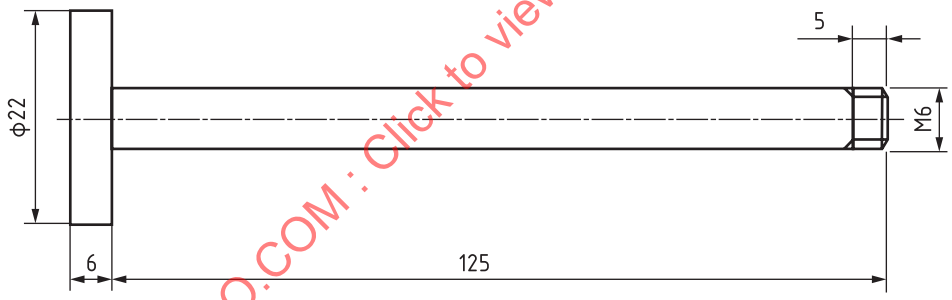
Key

1 multi-part test apparatus aligned with specimen set to universal testing machine
2 holders for tooth and material
A specimen
B tooth cup (holder for tooth)
C coupling for material rod/alignment rod
D material rod (holder for material)

NOTE Figure A.1 is made from that described in ISO/TR 11405:1994.[3)]

Figure A.1 — Jigs and alignment for Kemper and Killian's test method

Dimensions in millimetres



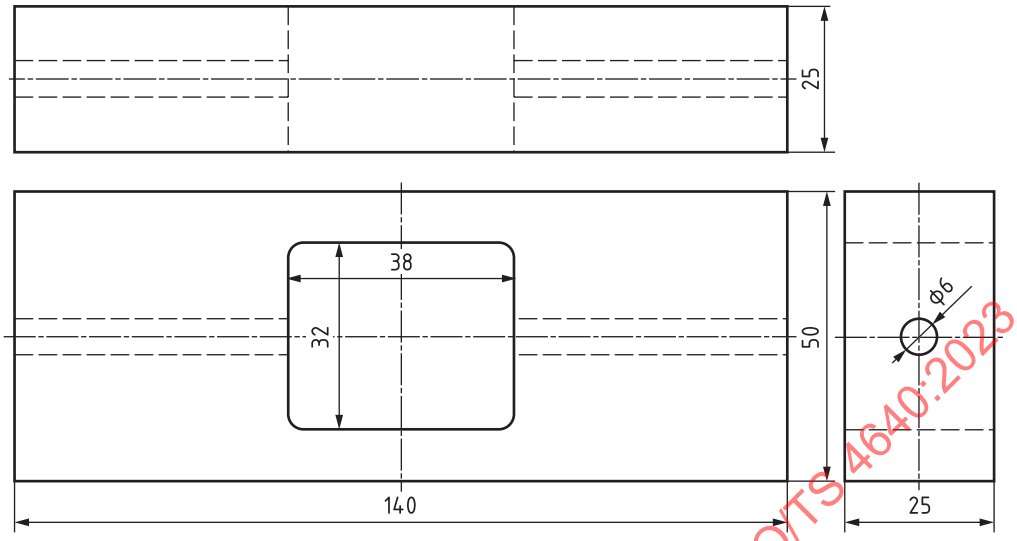
NOTE 1 Drawing of the longitudinal section.

NOTE 2 "Φ": diameter, "M6": metric screw of 6 mm in diameter, "— .— .— ": centre line.

a) Alignment rod for measurement block

3) Cancelled and replaced by ISO/TS 11405:2015.

Dimensions in millimetres



NOTE 3 The drawing on the top is a longitudinal section of the block, the one on the bottom is a top view, and the one to the right is a side view.

NOTE 4 " ----------": inner surface.

b) Measurement alignment block

NOTE 5 Two pieces of the alignment rods described in Figure A.2 a) are necessary.

Figure A.2 — A set of alignment and rods for the connection to the universal testing machine

A.2.1.2 Features in merit

a) The device for aligning two moulds, one of which a tooth is embedded in and the other, a resin composite is embedded in, is attached to a universal testing machine. The device is designed so that a tensile load is generated in a direction precisely perpendicular to the bonded surface.

b) Light-transparent moulds are included in the kit. The mould can also be used for photo-polymerisable (light-curable) materials.

A.2.2 Bencor's test method

A.2.2.1 Jigs and alignment

a) Based on a commercially available apparatus (Bencor Multi-T testing device) (see Figure A.3) for making specimens and performing tensile tests under controlled conditions (alignment).

b) Bencor's test method uses partly the same principles as described in the previous test (A.2.1).

A.2.2.2 Feature in merit

The material holder, in the shape of cylindrical tube made of metal with both ends of it are open, allows the use of light-curing materials.[7]

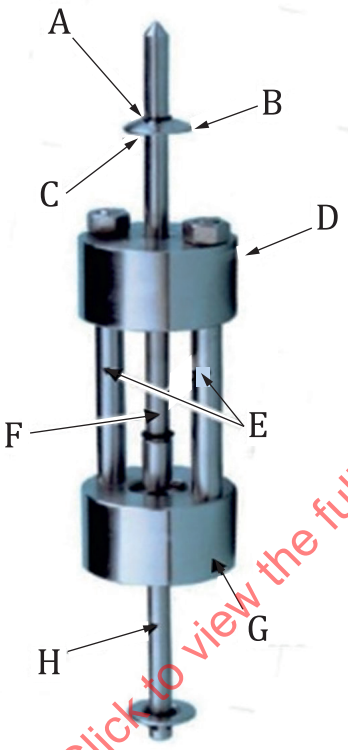
A.2.2.3 Preparation and fixing of test specimen

a) Appropriate lubricant shall be applied on the surface of the mould in order to reduce the frictional force between the mould and the material filled in the mould.

b) Avoid over-filling the materials on the upper edge of the mould not to stick the polymerised material to the upper edge of the mould.

c) Fix an appropriate jig (i.e. small metal screw with head of appropriate size) into the material (uncured) to hold the material cured to the tensioning rod of the tensile testing equipment.

d) Insert a U-shaped pin made of metal between the upper edge of the mould and under the face of the head of the jig described in c).



Key

A C-clip
B universal support
C O-ring
D head
E pillars
F active rod
G base
H suspension rod

NOTE A test specimen (tooth adhered to resin composite with the adhesive tested) is set between the rods indicated by key "F" and key "H".

Figure A.3 — A set of alignment and rods (to be connected to the universal testing machine)

A.2.3 Dumbbell test

A.2.3.1 Shape of the adhered specimen

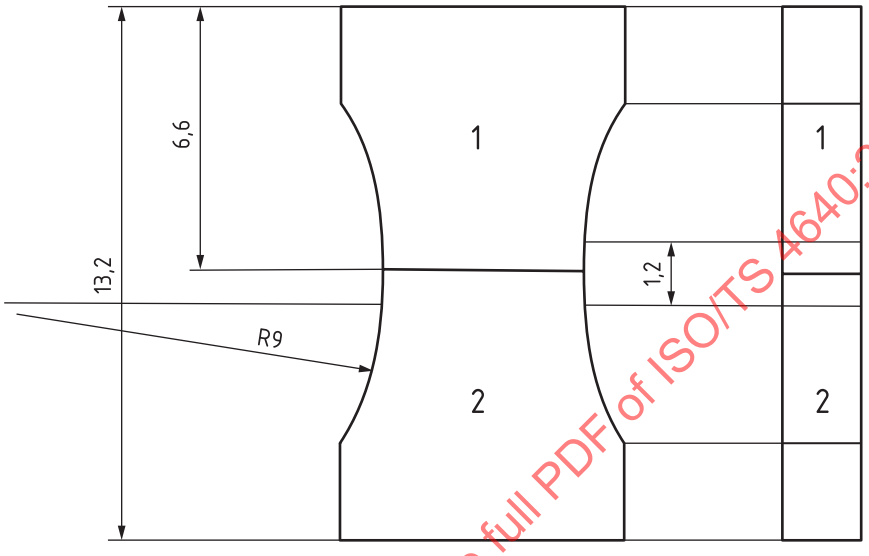
A dumbbell shaped specimen with a rectangular cross-section of bonded area, cut from a larger tooth/adhesive composite adhered specimen (see Figures A.4 and A.5).

A.2.3.2 Features in merit

a) The specimen prepared above allows a good control of the bonding area and guides the fracture to the adhesive interface.

b) Specimens limited to a 3 mm × 2 mm of bonding area, termed “Mini-Dumbbell”, seem to give more information on the bonding surfaces and the bonding mechanism.[8],[9]

Dimensions in millimetres



Key

1 Poly (methyl methacrylate) (PMMA)

2 dentine

Figure A.4 — Dimension of “Mini-Dumbbell” specimen